

## PATENT SPECIFICATION

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## (54) MAGNESIUM-THERMIC REDUCTION OF CHLORIDES

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ations organised and existing under the laws  
of the Union of Soviet Socialist Republics,  
do hereby declare the invention, for which  
we pray that a patent may be granted to us,  
and the method by which it is to be  
performed, to be particularly described in  
and by the following statement:

The present invention relates to a method  
and apparatus for producing metals by  
magnesium-thermic reduction of chlorides,  
especially rare metals such as titanium,  
zirconium, and hafnium. The invention is  
well suited for application in the production  
of rare metals to be further used as compo-  
nents of various alloys.

The invention provides a method of pro-  
ducing a metal by magnesium-thermic re-  
duction of its chloride, comprising charging  
the metal chloride and magnesium into a  
reaction chamber sealed with a cover and  
introduced into a heating furnace, the metal  
chloride and magnesium being charged  
through an opening in the cover, and, on  
completion of the reduction process, closing  
the opening by means of a fusible closure,  
connecting a condenser to the reaction  
chamber to form a vacuum separator, intro-  
ducing the vacuum separator into a vacuum  
furnace, and removing the fusible closure by  
evacuating the separator, so that vacuum

refining of the reduced metal then occurs.

This method makes it possible to intensify  
the reduction process owing to the fact that  
the fusible closure is fitted only on comple-  
tion of the reduction process, which also  
simplifies the reduction chamber construc-  
tion and improves its operating conditions.

In addition, the method also makes it  
possible to intensify the vacuum refining  
process due to the possibility of destroying  
the fusible closure precisely at the moment  
of initial phase of vigorous sublimation of  
magnesium and magnesium chloride from  
the reaction mass, which enhances the  
operating efficiency of the equipment used  
and diminishes power input required.

It is advisable that the temperature of the  
reaction mass before its introduction in the  
vacuum chamber be maintained above the  
freezing point of magnesium and below that  
reached during reduction. It is thus possible  
to preserve a considerable amount of heat,  
accumulated by the reaction mass in the  
course of reduction process and required for  
the vacuum refining process, and thereby to  
decrease the power input.

The invention also provides apparatus for  
use in the above method, comprising a  
reaction chamber in the form of a shell  
provided with a cover having a centrally  
disposed opening closed with a fusible clo-  
sure: a removable condenser in the form of  
a shell tightly connected to the reaction  
chamber to form therewith a vacuum separ-  
ator; and a heat shield arranged in the  
condenser and comprising at least two coa-  
xial cylinder members interconnected in a  
fluid-tight manner, the outer cylinder mem-  
ber bearing against a tapered portion of the  
cover surrounding the fusible closure.

The heat shield provides for effective thermal insulation of the fusible closure from the cooled parts of the vacuum separator, as well as for the timely destruction of the fusible closure during vacuum refining.

It is advantageous that the cover of the reaction chamber should be formed with an upwardly tapering portion whereupon is mounted the heat shield enveloping the fusible closure. This provides for coaxial arrangement of the heat shield with respect to the fusible closure at the place where it should most be thermally insulated from cooled parts of the vacuum separator.

It is likewise advantageous that the heat shield be provided with a thermally insulating layer adjacent to the interior surface of the outer cylinder member. Such a disposition of the thermally insulating layer makes it possible to improve the insulating performance of the heat shield and diminish the heat flow passing from the fusible closure towards the cooled parts.

It is most preferable that the height to width ratio of the shells of the reaction chamber and the condenser be more than 2 to 1. This ratio of the construction dimensions enables an appropriate disposition of the heat shield with respect to the fusible closure and the cooled parts, the shield partially extending into the interior of the reaction chamber shell and partially into the interior of the condenser shell, with the reaction chamber volume and condensing area of the condenser remaining unaltered.

One embodiment of the present invention will now be explained in detail, by way of example, with reference to the accompanying drawings, in which:

Figure 1 is a longitudinal sectional view of a reaction chamber; and

Figure 2 is a longitudinal sectional view of a vacuum separator on a reduced scale.

The reaction chamber shown in Figure 1 comprises a shell 1 provided with a means 2 for discharging magnesium chloride. The chamber is sealed by means of a concave cover 3 having an upwardly tapered portion 4. Mounted on a central pipe 5 of the cover 3 is a pipe 6 adapted for charging magnesium and the metal chloride to be reduced.

Shown in Figure 2 is a vacuum separator comprising the shell 1 and a similar shell 7, the shells forming a closed space and each having the height to diameter ratio of more than 2 to 1. The shell 7 serves as a condenser. A heat shield 8 having a heat-insulating layer 9 enveloping a fusible plug 10 rests on the upwardly tapering portion 4 of the cover 3. The heat shield 8 consists of at least two coaxially arranged cylinder members interconnected in a fluid-tight manner. The condenser 7 is connected to an evacuation means through a branch pipe 11. It is sealed to the shell 1 with sealing means 15,

and is surrounded by a cooling jacket 13 provided with an outlet pipe 14. The bottom of the shell 1 is sealed with an element 12.

The above-described apparatus operates in the following manner.

First, connected in a fluid-tight manner to the central pipe 5 is the pipe 6 provided with branch pipes. The shell 1 is tightly closed by the cover 3, and the means 2 for discharging magnesium chloride is welded to the shell. The reaction chamber thus formed is evacuated, filled with inert gas, and heated within a furnace to a temperature of 750 to 900°C. Thereafter, magnesium and the metal chloride to be reduced are charged through the respective branch pipes of the pipe 6 into the chamber thereby to result in reduction reaction.

On completion of the reduction reaction the reaction chamber is removed from the furnace and arranged in a thermally insulated enclosure (not shown) wherein the discharging means 2 is cut off to be replaced by the sealing element 12, which is fixed by welding; the pipe 6 is removed to be replaced by the fusible closure 10. The heat shield 8 is then mounted on the tapered portion 4 of the cover 3 and is arranged so as to be coaxial with the fusible closure 10. Next the shell 1 is tightly connected by the sealing means 15 to the condenser shell 7 whereupon is mounted the water jacket 13. The thus assembled vacuum separator is placed in a vacuum furnace and connected to an evacuating means through the branch pipe 11. Delivered into or directly on the surface of the jacket 13, surrounding the condenser shell 7, is a cooling fluid, e.g. water, which is discharged through the outlet pipe 14. Then the furnace heaters are switched on.

When a given temperature is reached within the furnace, the condenser is evacuated, whereby the fusible closure 10 is destroyed within a short period of time to give rise to vigorous evaporation of magnesium and magnesium chloride from the mass in the reaction chamber. The vacuum separation process is carried out at a temperature of 1000 to 1020°C at the walls of the shell 1. The completion of the vacuum separation process is determined by any conventional means. The apparatus containing the refined metal is then cooled and disassembled. The magnesium condensate in the shell 7 can be re-used in a succeeding reduction reaction where the shell 7 is used as the shell of a reaction chamber. All the apparatus parts and units undergo cleaning, washing, and drying before each operating cycle.

The above-described procedure, compared with conventional procedures, makes it possible to diminish power input and enhance operating efficiency, as well as reduce the duration of the vacuum separa-

tion process by 4 to 10 times depending upon the dimensions of the equipment being used.

Given below are two examples of the above-described procedures.

#### Example 1

Charged into the reaction chamber, whose steel shell 1 has a height to diameter ratio of more than 2 to 1, is molten magnesium in an amount of 3330 kg. The reaction chamber is then held at a temperature of 800 to 850°C while  $\text{TiCl}_4$  is continuously fed in. The magnesium chloride formed is discharged intermittently in portions of 500 kg each. After magnesium is used, the supply of  $\text{TiCl}_4$  is decreased by 60 per cent. The reaction mass which remains in the chamber has the following composition: titanium, about 2000 kg; magnesium, 1300 kg; and magnesium chloride, 500 kg.

The reaction chamber is then removed from the furnace and placed within the thermally insulated enclosure. The discharging means is cut off and a steel sealing element 12 is welded in its place. The pipe 6 for feeding the reagents is detached and the fusible cover 10 is set on the central pipe of the cover 3. The condenser is then tightly connected to the reaction chamber. The thus assembled vacuum separator is placed in the vacuum furnace and connected to the evacuation means the cooling jacket 13 of the condenser being connected to a water supply means. The furnace heaters are then switched on, and when the temperature in the furnace reaches 850 to 900°C the reaction chamber and the condenser are concurrently evacuated to thereby cause destruction of the fusible closure. Thereafter, the vacuum separation process proceeds at a temperature within the furnace of 980 to 1020°C.

The resultant refined titanium, obtained in the form of a block of sponge, amounted to about 2000 kg, and the resultant condensate was found to contain 1300 kg of magnesium and 500 kg of magnesium chloride.

#### Example 2

Charged into the reaction chamber, whose steel shell 1 has a height to diameter ratio of more than 2 to 1, is molten magnesium in an amount of 1000 kg. The reaction chamber is kept at a temperature of 800 to 870°C as zirconium chloride is continuously fed in. The magnesium chloride formed is intermittently discharged in portions of 200 kg each. After magnesium is used, the supply of zirconium chloride is decreased by 60 per cent. The reaction mass which remains within the reaction chamber has the following composition: zirconium,

about 800 kg; magnesium, 500 kg; and magnesium chloride, 120 kg.

The reaction chamber is then removed from the furnace and placed within the thermally insulated enclosure. The discharging means 2 is cut off and a steel sealing element 12 is welded in its place. The pipe 6 for feeding reagents is detached and the fusible closure 10 is set on the central 5 of the cover 3. The heat shield 8 is mounted on the cover, and the condenser is tightly connected to the reaction chamber. The thus assembled vacuum separator is placed in the vacuum furnace and connected to the evacuation means, the condenser being connected to a water supply means. The furnace heaters are then switched on, and when the temperature in the furnace reaches 850 to 900° the reaction chamber and condenser are concurrently evacuated to thereby cause destruction of the fusible closure. Thereafter, the vacuum separation process proceeds, with the temperature within the furnace ranging from 900 to 960°C.

The resultant refined zirconium, obtained in the form of a block, amounts to about 800 kg, and the resultant condensate was found to contain 500 kg of magnesium, and 120 kg of magnesium chloride.

Titanium is obtained in the form of metallic sponge because during the evacuation process there is active reduction of titanium chlorides having the lowest valencies, formed at the surface of the reaction mass during the final stage of reduction.

The above-described method makes it possible to diminish specific power input and enhance operating efficiency of the vacuum separation process by avoidance of the inefficient stage of heating solid-phase products which have, in comparison with liquids, poor thermal characteristics, as well as by the conservation of heat accumulated by the reaction mass during reduction process.

#### WHAT WE CLAIM IS:-

1. A method of producing a metal by magnesium-thermic reduction of its chloride, comprising charging the metal chloride and magnesium into a reaction chamber sealed with a cover and introduced into a heating furnace, the metal chloride and magnesium being charged through an opening in the cover, and on completion of the reduction process, closing the opening by means of a fusible closure, connecting a condenser to the reaction chamber to form a vacuum furnace, and removing the fusible closure by evacuating the separator, so that vacuum refining of the reduced metal then occurs.

2. A method as claimed in claim 1, wherein, after completion of the reduction process, the temperature of the reaction

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mass is maintained above the freezing point of magnesium and below that pertaining during reduction, until the vacuum separator is introduced into the vacuum furnace.

- 5     3. Apparatus for use in the method of claim 1, comprising a reaction chamber in the form of a shell provided with a cover having a centrally disposed opening closed with a fusible closure; a removable condenser in the form of a shell tightly connected to the reaction chamber to form therewith a vacuum separator; and a heat shield arranged in the condenser and comprising at least two coaxial cylinder members interconnected in a fluid-tight manner, the outer cylinder member bearing against a tapered portion of the cover surrounding the fusible closure.

- 10     4. Apparatus as claimed in claim 3, wherein the said portion of the cover is upwardly tapered.

- 15     5. Apparatus as claimed in claim 3 or 4, wherein the heat shield has a heat-insulating layer adjacent to the upper surface of the said one cylinder member.

- 20     6. Apparatus as claimed in claim 3, wherein the shells each have a height to width ratio of more than 2 to 1.

- 25     7. A method as claimed in claim 1, substantially as described herein with reference to the accompanying drawings.

- 30     8. A method as claimed in claim 7, substantially as described in Example 1 or Example 2.

- 35     9. Apparatus as claimed in claim 3, substantially as described herein with reference to, and as shown in, Figure 2 of the accompanying drawings.

- 40     10. Metal produced by a method according to any of claims 1, 2, 7, and 8.

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COMPLETE SPECIFICATION

1 SHEET

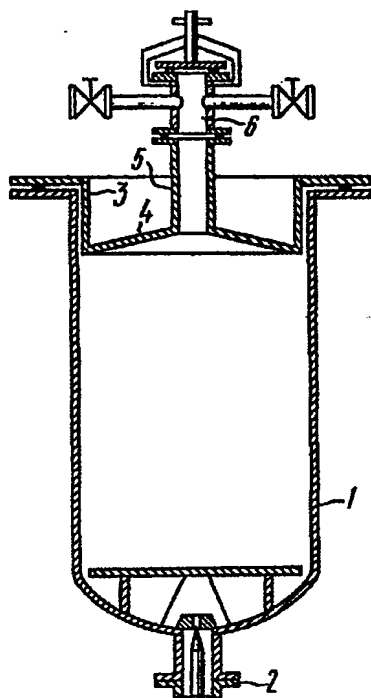
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FIG. 1

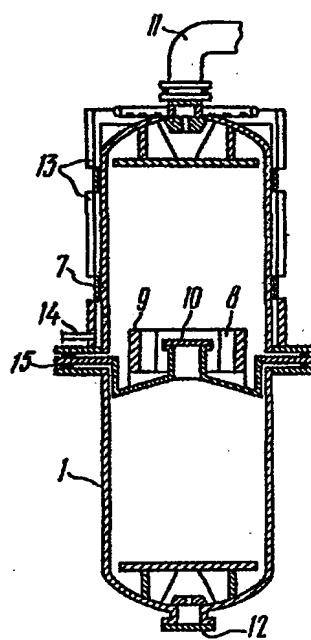


FIG. 2

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